National Environmental Laboratory **Accreditation** Conference

nelac

Quality Systems

Proposed Changes

January 12, 1998

5.1 SCOPE

. . . .

b) This standard includes additional requirements and information for assessing competence or for determining compliance by the organization or accrediting authority granting the recognition (or approval).

If more stringent standards or requirements are included in a mandated test method or by regulation, the laboratory shall demonstrate that such requirements are met. (See the supplemental accreditation requirements in Section 1.9.2.)

5.4.2 Organization

. . . .

h) ~~where applicable, nominate deputies in case of absence of the technical director or quality assurance officer and shall accomplish this by having contingency plans in the event that either the technical director or quality assurance officer is absent;~~

nominate deputies in case of absence of the technical director and/or quality assurance officer;

5.5.2 Quality Manual

. . . .

r) procedures for protecting confidentiality~~,~~ ~~and~~ proprietary rights, and national security concerns;

5.5.4 Essential Quality Control Procedures

. . . .

b) All quality control measures shall be assessed and evaluated on an on-going basis, and quality control acceptance ~~limits~~ criteria shall be used to determine the useability of the data (See Appendix D).

c) The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exist. (See 5.11.2, Sample Acceptance Policy.)

5.6.2 Laboratory Management Responsibilities

In addition to 5.4.2.d, the laboratory management shall be responsible for:

. . . .

c) Ensuring that the training of its personnel is kept up-to-date by the following:

. . . .

3) ~~Analyst training shall be considered up-to-date when documentation in the files indicate acceptable performance of a blind sample (singly blind to the analyst) at least once per year and a certification that technical personnel have read, understood and agreed to perform the most recent version of the method, the approved method (if applicable) or standard operating procedure;~~
Analyst training shall be considered up to date if the employee file contains a certification that technical personnel have read, understood and agreed to perform the most recent version of the method, the approved method (if applicable) or standard operating procedure, and documentation of continued proficiency by at least one of the following:
. Acceptable performance of a blind sample (single blind to the analyst) at least once per year;
ii. Analysis of another initial demonstration of method performance;
iii. Successful analysis of a blind performance sample on a similar method using the same technology (e.g., GC/MS volatiles by purge and trap for 524.2, 624 or 8260) would only require documentation for one of the methods.
iv. Control chart with at least four consecutive laboratory control samples with acceptable levels of precision and accuracy within the past year;
. ~~Analyst's technique reviewed or audited for adherence to method requirements by an external agency, an internal auditor, or supervisor; or~~
~~vi.~~ Analysis of authentic samples that have been analyzed by a proficient analyst with statistically identical results.

5.7 PHYSICAL FACILITIES - ACCOMMODATION AND ENVIRONMENT

5.7.1 Environment

c) The laboratory shall provide ~~facilities~~ for the effective monitoring, control and recording of environmental conditions as appropriate. Attention shall be paid, for example, to biological sterility, dust, electromagnetic interference, humidity, mains voltage, temperature, and sound and vibration levels~~, as appropriate to the calibrations or tests concerned~~.

5.9.3 Reference Standards

a) Reference standards of measurement held by the laboratory (such as Class S or equivalent weights or traceable thermometers) shall be used for calibration only and for no other purpose, unless it can be demonstrated that their performance as reference standards ~~has~~ have not been invalidated. Reference standards of measurement shall be calibrated by a body that can provide, where possible, traceability to a national standard of measurement.

b) There shall be a program of calibration and verification for reference standards.

c) ~~Where relevant, reference standards and measuring and testing equipment shall be subjected to in-service checks between calibrations and verifications.~~ Reference materials shall, where possible, be traceable to national or international standards of measurement, or to national or international standard reference materials.

5.9.4.2.1 Analytical Support Equipment

. . . .

b) calibrated or verified at least annually, using NIST traceable references when available, over the entire range of ~~in which the equipment is~~ used. The results of such calibration shall be within ± the manufacturer's published specifications. If the calibration/verification is not within the manufacturer's published specifications: ~~stated sensitivity or:~~

5.9.4.4.2 Continuing Calibration Verification

. . . .

a) These standards shall be analyzed at a frequency of 5% or every 12 hours whichever is more frequent and may be the standards used in the original calibration curve or standards from another source. The frequency shall be increased if the instrument consistently drifts outside acceptance ~~ble limits~~ criteria before the next calibration.

c) A new curve shall be run if two back-to-back runs of one continuing calibration check is outside acceptance ~~ble~~ criteria ~~limits~~. When the continuing calibration [check] acceptance criteria ~~limit~~ are ~~is~~ exceeded high (i.e., high bias), and there are non-detects for the corresponding analyte in all environmental samples associated with the continuing calibration check, then those non-detects may be reported, otherwise the samples affected by the unacceptable check shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Additional sample analysis shall not occur until a new calibration curve is established and verified.

5.10.1.2 Laboratory Method Manual(s)

. . . .

b) . . . .

1) identification of the test method~~ and where applicable, the analyte name with qualifier (the qualifier is a word, phrase or number that better identifies the method; e.g., "Iron, Total", or "Chloride, Automated Ferricyanide", or "Our Lab. Method SOP No. 101");~~
2) applicable matrix or matrices;
3) method detection limit;
4) scope and application, including components to be analyzed;

5.10.2 Test Methods

a) The laboratory shall use appropriate methods and procedures for all tests and related activities within its responsibility (including sampling, handling,

transport and storage, preparation of items~~, estimation of uncertainty of measurement~~ and analysis of test data). The method and procedures shall be consistent with the accuracy required, and with any standard specifications relevant to the calibrations or tests concerned.

5.10.5 Documentation and Labeling of Standards and Reagents

. . . .

a) The laboratory shall retain records~~, such as manufacturer's statement of purity, of the origin, purity and traceability of all standards (including balance weights and thermometers). Records~~ for all standards ~~shall include~~ including the manufacturer/vendor, the manufacturer's Certificate of Analysis or purity (if supplied), the date of receipt, recommended storage conditions, and ~~if applicable, the date of opening and~~ an expiration date after which the material shall not be used.

b) Original ~~reagent~~ containers shall be labeled with ~~the date opened and~~ an expiration/disposal date.

c) Detailed records shall be maintained on reagent and standard preparation. These records shall indicate traceability to purchased stocks or neat compounds, reference to ~~and must include~~ the method of preparation, date of preparation, and preparer's initials.

~~d) Where calibrations do not include the generation of a calibration curve, such as thermometers, balances, or titrations, records shall indicate the calibration date and type (balance weight, thermometer serial number, primary standard concentration) of calibration standard that was used.~~

d~~e~~) All prepared reagents and standards must be uniquely identified and the contents shall be clearly identified with preparation date, concentration(s) and preparer's initials.

5.11.2 Sample Acceptance Policy

. . . .

c) Use of appropriate sample containers~~;~~

d) Adherence to specified holding times; ~~and~~

e) Adequate sample volume. Sufficient sample volume must be available to perform the necessary tests; and

f) Samples which show signs of damage or contamination.

5.11.4 Storage Conditions

The laboratory shall have documented procedures and appropriate facilities to avoid deterioration, ~~,~~ contamination, or damage to the sample~~;~~ during storage, handling, preparation, and testing; any relevant

. . . .

2) Samples shall be stored away from all standards, reagents, food and other potentially contaminating sources, including highly contaminated samples.

5.11.5 Sample Disposal

The laboratory shall have standard operating procedures for the disposal of samples, digestates, leachates and extracts or other sample preparation products~~, including all provisions necessary to protect the integrity of the laboratory~~.

5.12.2 Records Management and Storage

. . . .

b) All records, including those specified in 5.12.3 and 5.12.4, ~~of an organization that are pertinent to a specified project~~ shall be retained for a minimum of five years~~ unless otherwise designated for a longer period of time in another regulation~~. ~~The records specified in 5.12.3 and 5.12.4 shall be retained.~~ All information ~~hardware and software~~ necessary for the historical reconstruction of data must be maintained by the laboratory. Records which are only stored on electronic media must be supported by the hardware and software necessary for their retrieval.

5.12.4.1 Basic Requirements

. . . .

h) ~~If samples are shipped, the shipping container shall be sealed in such a manner so that tampering by unauthorized personnel is immediately evident~~ If samples are submitted with sample custody seals, and any seals are not intact, the lab shall note this in the chain of custody.

5.13 LABORATORY REPORT FORMAT AND CONTENTS

The results of each test, or series of tests carried out by the laboratory shall be reported accurately, clearly, unambiguously and objectively~~, in accordance with any instructions in the test methods~~. The results shall normally be reported in a test report and shall include all

. . . .

a) . . . .

6) ~~where relevant, characterization and condition of the sample~~ identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature;

9) ~~where relevant~~ when the laboratory collected the sample, reference to sampling procedure;

10) any deviations from (such as failed quality control), additions to or exclusions from the test method (such as environmental conditions), and any other information relevant to a specific test, such as environmental conditions including the use of relevant data qualifiers and their meaning;

11) ~~measurements, examinations and derived results, supported by tables, graphs, sketches and photographs as appropriate, and any failures (such as failed quality control) identified. Where relevant, include a description of the transformations, calculations, or operations performed on the data, a summary and analysis of the data. Where applicable,~~ identification of whether data ~~is~~ are calculated on a dry weight or wet weight basis; identification of the reporting units such as μg/l or mg/kg~~.~~ and for Whole Effluent Toxicity,

identification of the statistical package used to provide data.

12) ~~where relevant~~ when required ~~by the client or by a regulatory agency~~, a statement of the estimated uncertainty of the test result~~; such as a value reported below the limit of quantitation~~;

~~In situations where required by the client or regulatory agency, this information shall be provided. It may be required of laboratories involved in analyses, where there is an uncertainty associated with detection limits.~~

14) ~~where relevant~~ at the laboratory's discretion, a statement to the effect that the results relate only to the items tested or to the sample as received by the laboratory;

15) ~~where relevant~~ at the laboratory's discretion, a statement that the certificate or report shall not be reproduced except in full, without the written approval of the laboratory; ~~and~~

16) ~~where relevant,~~ when reported clear identification of all data provided by outside sources, such as air temperature or ambient water temperature; and~~.~~

17) clear identification of numerical results with values below 3.18 times the MDL (10 times the standard deviation as determined by the method detection limit study).

Appendix B - DEFINITIONS FOR QUALITY SYSTEMS

Acceptance ~~ble~~ Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Analytical Detection Limit (LD): ~~the smallest amount of an analyte that can be distinguished in a sample by a given measurement procedure throughout a given (e.g., 0.95) confidence interval.~~ the minimum concentration of an analyte, that, in a given matrix and with a specific method, has a 99% probability of being identified, qualitatively or quantitatively measured, and reported to be greater than zero [The analytical detection limit shall be established initially and verified annually for each method and sample matrix.]

Analytical Reagent (AR) Grade: designation for the high purity of certain chemical reagents and solvents given the American Chemical Society. (Quality Systems)

Assessor Body: the organization that actually executes the accreditation process, i.e., receives and reviews accreditation applications, reviews QA documents, reviews proficiency testing results, surveys the site, etc., whether EPA, the state, or contracted private party. (NELAP)

~~**Accuracy**: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).~~

~~**Analytical Reagent (AR) Grade**: designation for the high purity of certain chemical reagents and solvents given the American Chemical Society. (Quality Systems)~~

~~**Calibration Method:** defined technical procedure for performing a calibration.~~

Detection Limit: the lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated degree of confidence. See Method Detection Limit.

~~**Instrument Blank**: a clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).~~

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank ~~quality control sample~~): an uncontaminated sample matrix spiked with known amounts of analytes from a source independent of the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Limit of Detection (LOD): the lowest concentration level that can be determined (by a single analysis and with a defined level of confidence) to be statistically different from a blank. (Analytical Chemistry, 55, p.2217, December 1983, modified) See also Method Detection Limit.

Proficiency Test Sample (PT): a sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria ~~performance limits~~. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

~~**Quality Control Sample**: an uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).~~

Quantitation Limits: the maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. Quantitation limit, for the purposes of NELAC, is defined as three times the MDL, by convention.

~~**Reagent Blank (method reagent blank)**: a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).~~

~~**Replicate Analyses:** the measurements of the variable of interest performed identically on two or more subsamples of the same sample within a short time interval.~~

~~**Sample Duplicate**: two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).~~

~~**Systems Audit (also Technical Systems Audit):** a thorough, systematic on-site, qualitative review of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system.~~

~~**Technical Analyst**: the designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent Quality Controls to meet the required level of quality.~~

Appendix D - ESSENTIAL QUALITY CONTROL REQUIREMENTS

The quality control protocols specified by the laboratory's method manual (5.10.1.2) shall be followed. The laboratory shall ensure that the essential standards outlined in Appendix D are incorporated into their method manuals

All quality control measures shall be assessed and evaluated on an on-going basis and quality control acceptance ~~limits~~ criteria shall be used to determine the validity of the data. The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exists.

D.1 CHEMICAL TESTING

D.1.1 Positive and Negative Controls

. . . .

b) Positive Controls

. . . .

3) Surrogates - Surrogate compounds must be added to all samples, standards, and blanks, whenever possible, for all organic chromatography methods. Poor surrogate recovery may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

D.1.4 Method Detection Limits

. . . .

b) The detection limit shall be initially determined for the compounds of interest in each method in a clean matrix appropriate to the test method (such as laboratory pure reagent water or Ottawa sand) or the matrix of interest (see definition of matrix).

c) ~~The laboratory must verify that the MDL is at least three (3) times less than the laboratory reporting limit.~~ Laboratories shall assign numerical or quantitative values to all results greater than three times the MDL. All quantitatively reported results (i.e., those greater than three times the MDL) shall be bracketed by calibration standards. Numerical values may also be assigned to results lower than three times the MDL, but these must be

identified and be recognizable as having lower associated confidence levels.

D.3.3 Method Evaluation

a) In order to demonstrate the suitability of a method for ~~specified purpose~~ an intended purpose, the laboratory shall demonstrate and document its ability to meet acceptance criteria either specified by the method or by the client's requirements. Acceptance criteria must meet or exceed client requirements and must demonstrate that the method provides correct/expected results with respect to specified detection capabilities, ~~establish, through method validation, a set of acceptance criteria for the performance characteristics of the method unless such criteria are specified by the method. These criteria must demonstrate that the method provides a correct/expected result with respect to specified~~ acceptance criteria ~~limits of detection,~~ selectivity, repeatability, sensitivity and reproducibility.

. . . .

2) Qualitative microbiological test methods in which the response is expressed in terms of presence/absence, shall be validated by estimating, if possible, the specificity, relative trueness, positive deviation, negative deviation, repeatability, reproducibility and the minimum detection capability ~~limit of determination~~ within a defined variability. The differences due to the matrices must be taken into account when testing different sample types.

Figure D-1. USE OF REFERENCE CULTURES (BACTERIA)

Flow Chart

Reference culture from source recognized by NELAC

Culture once
Appropriate Purity Checks and Biochemical Tests ~~as Appropriate~~

Reference Stocks
Retained under specific Conditions:
Freeze dried, liquid nitrogen storage, deep frozen or other storage means under specified conditions and storage times/

~~Purity Checks and Biochemical Tests as Appropriate~~

Thaw/Reconstitute
~~Purity Checks and Biochemical Tests as Appropriate~~

Working Stocks
Maintained under specific conditions and storage times

Regular/Daily Quality Controls

Appendix E - PERFORMANCE BASED MEASUREMENT SYSTEM

RESERVED - The information presented here is the most recent EMMC Workgroup draft, and is provided for information only.

E.1 Checklist Overview

The Checklists present consensus among EPA's programs on performance "categories" that allow use of the same Checklists across the Agency's various programs/projects. The Checklists may be applied to screening and field techniques as well as traditional laboratory procedures.

Implementation of the Checklists is intended to be program-specific and a category that does not apply within a specific EPA program or project will be indicated by NA (not applicable). Criteria for a specific EPA program or project are to be filled in under the "Performance Criteria" column; e.g., an Office of Water Reference Method may specify 20% RSD or a correlation coefficient of 0.995 for the category that specifies calibration linearity, whereas an Office of Solid Waste project may specify a Measurement Quality Objective of 12% RSD or a correlation coefficient of 0.998 for this category.

For each EA program or project, the checklists are to be completed for each matrix within each medium for which performance is demonstrated.

Each completed Checklist must be retained on file at the laboratory that uses the performance-based method (PBM) or method modification and must be submitted to the appropriate regulatory authority upon request to support analysis of those samples to which the PBM or modified method was applied.

E.1.1 Header

Each page of the checklist contains six lines of header information, consisting of:

a) Date: enter the date that the checklist was completed and associated samples were collected.

b) Laboratory Name & Address: If the method is being employed by a commercial contract laboratory on behalf of one or more applicable clients, enter the name of the laboratory if possible followed by a listing of the

appropriate clients from which the samples were collected).

c) Discharge Point ID, where applicable.

d) Facility Name: enter the name of the water treatment facility, system, or regulated facility or other program/project specified entity where the facility maintains an on-site analytical laboratory.

e) EPA Program & Applicable Regulation: enter the name of the Agency program or project to whom the results will be reported, or under the auspices of which the data are collected, e.g., “CAA” for Clean Air Act testing/monitoring and “SDWA” for analyses associated with the Safe Drinking Water Act.

f) Medium: enter the type of environmental sample, e.g., water--NOTE a separate checklist should be prepared for each matrix, e.g., for checklists associated with performance-based methods for SDWA, enter Drinking Water as the matrix type. As the evaluations of a performance-based method will involve matrix-specific performance measures, a separate checklist would be prepared for each matrix. The medium is the environmental sample type to which the performance-based method applies, whereas the performance category matrix, appearing in the body of the checklists refers to the specific sample type within the Medium that was spiked, e.g., for Medium hazardous waste, the checklist category Matrix may be solvent waste.

g) Analyte, Class of Analytes, or Other Measured Parameters--CAS # where available: As many methods apply to a large number of analytes, it is not practical to list every analyte in this field, as indicated on the form, the class of analytes may be listed here, i.e., volatile organics. However, if such a classification is used, a separate list of analytes and their respective Chemical Abstract Service Registry Numbers (CAS #) must be attached to the checklist.

~~C.2~~ E.1.2 EPA PBMS Checklist for Initial Demonstration of Method Performance

The Initial Demonstration of Method Performance involves multiple spikes into a defined sample matrix (e.g.,

wastewater, paper plant effluent), to demonstrate that the Performance-based Method meets the Program or Project Performance Criteria based on the performance of established Reference Method or based on Measurement Quality Objectives (analytical portion of the Data Quality Objectives). This exercise is patterned after the Initial Demonstration of Capability in C.1 of this appendix.

Footnote #1 indicates that a detailed narrative description of the initial demonstration procedure is to be provided.

Footnote #2 For multi-analyte methods, enter “see attachment” and attach a list or table containing the analyte-specific performance criteria from the reference method or those needed to satisfy measurement quality objectives. Complete only one of the two columns. For multi-analyte methods it is suggested that the list also contain the information for the “Results Obtained” and Performance Specification Achieved” columns.

Footnote #3 indicates that if a reference method is the source of the performance criteria, the reference method should be appropriate for its intended application and the listed criteria should be fully consistent with that reference method. The reference method name and EPA number (where applicable) should be delineated.

There are 34 numbered entries in the body of the checklist--each program will indicate the performance categories which do not pertain to the application/project, e.g., by listing as NA ("Not Applicable") for the corresponding performance criteria.

#1. Written Method (addressing all elements in the EMMC format)

The details of the method used for analysis (and sampling, where applicable) should be described in a version of the method written in EMMC format. The EMMC method format includes the following sections: 1.0 Scope & Application; 2.0 Summary of Method; 3.0 Definitions; 4.0 Interferences; 5.0 Safety; 6.0 Equipment & Supplies; 7.0 Reagents & Standards; 8.0 Sample Collection, Preservation & Storage; 9.0 Quality Control; 10.0 Calibration & Standardization; 11.0 Procedure; 12.0 Data Analysis & Calculations; 13.0 Method Performance; 14.0 Pollution Prevention; 15.0 Waste Management; 16.0 References; 17.0 Tables, Diagrams, Flowcharts & Validation Data. While this format may differ from that used in standard operation procedures (SOPs) in a

given laboratory, the use of a consistent format is essential for the efficient and effective evaluation by inspectors, program and project managers/officers.

#2. Title, Number and date/revision of “Reference Method” if applicable.

For example Polychlorinated Dioxins and Furans, EPA Method 1613, Revision B, October, 1994.

#3. Copy of the reference method, if applicable, maintained at the facility.

A copy of the reference method should be available to all laboratory personnel, however, it need not be attached to the checklist itself.

#4. Differences between PBM and reference method attached, if applicable.

The laboratory should summarize the differences between the reference method and the performance-based method and attach this summary to the checklist. This summary should focus on significant differences in techniques (e.g., changes beyond the flexibility allowed in the reference method), not minor deviations such as the glassware used.

#5. Concentrations of calibration standards.

The range of the concentrations of materials used to establish the relationship between the response of the measurement system and analyte concentration. This range must bracket any action, decision or regulatory limit. In addition, this range must include the concentration range for which sample results are measured and reported.

#6. % RSD or Slope/Correlation Coefficient of Calibration Regression.

This performance category refers to quantitative measures describing the relationship between the amount of material introduced into the measurement system and the response of the measurement system, such as an analytical instrument. A linear response is generally expected and is typically measured as either a linear regression (for inorganic analytes) or as the relative standard deviation (or coefficient of variation) of the response factors or calibration factors (for organic analytes). For example, traditional performance specifications consider any

regression line with a correlation coefficient (r) of 0.995 or greater as linear. Also, for organic analytes, a relative standard deviation (RSD) of 15% or less is often considered linear (RCRA). The calibration relationship is not necessarily limited to a linear relationship. However, it should be remembered if the Program/Project Office or Officer/Managers specifies other calibration relationships, e.g., quadratic fit, more calibration standards are generally necessary to establish accurately the calibration. If applicable, a calibration curve, graphical representation of the instrument response versus the concentration of the calibration standards, should be attached.

#7. Performance range tested (with units).

This range must reflect the actual range of sample concentrations that were tested and must include the concentration units. Since the procedures may include routine sample dilution or concentration, the performance range may be broader than the range of the concentrations of the calibration standards.

#8. Samples(s) used in initial demonstration have recommended preservative, where applicable. Sample(s) used in the initial demonstration should employ the recommended preservative, where applicable. Answer “yes” if the preservation in the reference method was used. If “no”, include a narrative description of the testing done to support use of the alternate preservation technique.

#9. Samples(s) used in the initial demonstration must be within the recommended holding times, where applicable.

Unless holding time (time from when a sample is collected until analysis) has been specifically evaluated, this entry should be taken directly from the reference method, where applicable or standard table. If holding time has been evaluated, include the study description and conclusions of that evaluation here, with a reference to the specific study description. The data must be attached.

#10. Interferences.

Enter information on any known or suspected interferences with the performance-based method. Such interferences are difficult to predict in many cases, but may be indicated by unacceptable spike recoveries in environmental matrices, especially when such recovery problems were not noted in testing a clean matrix such as reagent water. The

interferences associated with the reference method are to be indicated, as well as, the effect of these interferences on the performance-based method.

#11. Qualitative identification criteria used.

Enter all relevant criteria used for identification, including such items as retention time, spectral wavelengths and ion abundance ratios. If the instrumental techniques for these performance-based method are similar to a reference method, use the reference method as a guide when specifying identification criteria. If the list of criteria is lengthy, attach it on a separate sheet, and enter “see attached” for this item.

#12. Performance Evaluation Studies performed for analytes of interest, where available (last study sponsor and title last study number:).

Several EPA programs conduct periodic performance evaluation (PE) studies. Organizations outside of the Agency also may conduct such studies. Where available and applicable, enter the sponsor, title, and date of the most recent study in which the performance-based method was applied to the matrix of interest. A program/project may specify that a performance-based method be fully successful, i.e., within the PE study QC acceptance criteria. Where applicable, provide a listing of analytes for which the PE results were "not acceptable".

#13. Analysis of external reference material.

Enter the results of analyses on reference material from a source different from that used to prepare calibration standards (if available). This performance category is especially important if Performance Evaluation Studies are not available for the analytes of interest.

#14. Source of reference material.

Enter information, if applicable and available, for traceability of external reference materials used to verify the accuracy of the results, e.g., obtained from the National Institute of Science and Technology (NIST).

#15. Surrogates used, if applicable.

Enter the names of the surrogate compounds used. Surrogates are often used in analysis of organic analytes. Surrogates

may be added to samples prior to preparation, as a test of the entire analytical procedure. These compounds are typically brominated, fluorinated or isotopically labeled, with structural similarities to the analytes of interest. Target analytes of the method may be used as surrogates, if they can be demonstrated not to be present in the samples to be analyzed.

#16. Concentrations of surrogates, if applicable.

Enter the concentration of surrogates once spiked into the sample (i.e., final concentration).

#17. Recoveries of Surrogates appropriate to the proposed use, if applicable.

Enter the summary of the surrogate recovery limits; attach a detailed listing if more space is needed.

#18. Sample Preparation.

Enter preliminary procedures, e.g., digestion, distillation and/or extraction. A detailed listing may be attached if more space is needed.

#19. Clean-up Procedures.

Enter appropriate sample clean-up steps prior to the determinative step (instrumental analysis), e.g., GPC, copper, alumina treatment, etc.

#20. Method Blank Results.

A clean matrix (i.e., does not contain the analytes of interest) that is carried through the entire analytical procedure, including all sample handling, preparation, extraction, digestion, cleanup and instrumental procedures. The volume or weight of the blank should be the same as that used for sample analyses. The method blank is used to evaluate the concentrations of analytes that may be introduced into the samples as a result of background contamination in the laboratory. Enter the analyte/s and concentration measured in the blank.

#21. Matrix (reagent water, drinking water, sand, waste solid, ambient air, etc.).

Refers to the specific sample type within the broader Medium that was spiked, e.g., for Medium: Hazardous Waste an

example matrix spiked as part of the initial demonstration of method performance might be "solvent waste".

#22. Spiking System, appropriate to the method and application.

Enter the procedure by which a known amount of analyte/s ("spike") was added to the sample matrix. This may include the solvent that is employed and the technique to be employed (e.g., permeation tube, or volumetric pipet delivery techniques spiked onto a soil sample and allowed to equilibrate 1 day, etc.). Solid matrices and air are often difficult to spike and considerable detailed narrative may be necessary to delineate the procedure. For spikes into aqueous samples generally a water miscible solvent is needed.

#23. Spike concentrations (w/units corresponding to final sample concentration).

Enter the amount of the analyte/s ("spike") that was added to the sample matrix in terms of the final concentration in the sample.

#24. Source of spiking material.

Enter the organization or vendor from which the spiking material was obtained or how the spiking material was prepared. This should include specific identification information, e.g., lot#, catalogue number, etc.

#25. Number of Replicate Spikes.

The initial demonstration of method performance involves the analyses of replicate spikes into a defined sample matrix (category #21). Enter the number of such replicates. For example in the NPDES and SDWA programs, at least 4 replicates should be prepared and analyzed independently.

#26. Precision (analyte by analyte).

Precision is a measure of agreement among individual determinations. Statistical measures of precision include standard deviation, relative standard deviation or percent difference.

#27. Bias (analyte by analyte).

Bias refers to the systematic or persistent distortion of a measurement process which causes errors in one direction. Bias is often measured as the ratio of the measured value to the "true" value or nominal value. Bias is often (erroneously) used interchangeably with "accuracy", despite the fact that the two terms are complementary, that is, high "accuracy" implies low "bias", as well as good precision. Enter the name of the bias measure (% recovery, difference from true, etc.), and the numeric value with associated units for each analyte obtained for each analyte spiked in the initial demonstration procedure.

#28. Detection Limit (w/units; analyte by analyte), if applicable.

A general term for the lowest concentration at which an analyte can be detected and identified. There are various approaches to establishing detection limits ~~measures of detection~~ which include "Limit of Detection" and 'Method Detection Limit". Enter the approach used ~~detection measure~~ (e.g., MDL) and the analytical result with units for each analyte in the matrix (see #21).

This performance category is of importance when operating at extremely low concentrations. If the concentrations measured or the decisions to be made, e.g., action levels, are several orders of magnitude above these concentrations, the "quantitation level" should be entered.

#29. Confirmation of Detection Limit. if applicable.

In addition to spikes into the matrix of interest (see #21) it may be beneficial to perform the detection limit measurements in a clean matrix, e.g., laboratory pure water, air, sand, etc. Results of the spikes in the clean matrix are frequently available in the Agency’s published methods. Determining MDLs in a clean matrix using the performance-based method will allow a comparison to the MDLs published in the Agency methods.

This performance category is of importance when operating at extremely low concentrations. If the concentrations measured or the decisions to be made, e.g., action levels, are several orders of magnitude above these concentrations, the "quantitation level" should be entered.

Also, the detection limit technique may specify specific procedures to verify that the obtained limit is correct,

e.g., the "iterative process" detailed in the 40 CFR Part 136, Appendix B, MDL procedures.

#30. Quantitation Limit (w/ units; analyte by analyte).

The lowest concentration at which the analyte can be reported with sufficient certainty that an unqualified numeric value is reported. Approaches to establishing ~~Measures of~~ quantitation limits include the Minimum Level (ML), Interim Minimum Level (IML), Practical Quantitation Level (PQL), and Limit of Quantitation (LOQ). Enter the approach used to establish the ~~measure of~~ quantitation limits, and the corresponding units for each analyte appropriate to the intended application and a description of how hey were determined.

#31. Qualitative Confirmation.

Enter all relevant criteria used for identification, including such items as: retention time; use of second chromatographic column; use of second (different) analytical technique; spectral wavelengths, ion abundance ratios. If the instrumental techniques for the performance-based method are similar to those of a reference method, use the reference method as a guide when specifying confirmation criteria. If the list of criteria is lengthy, attach it on a separate sheet, and enter “see attached” for this item.

#32. Frequency of performance of Initial Demonstration:

Enter the frequency that the initial demonstration needs to be repeated.

#33-#34. Other Criteria.

Enter other necessary program/project specific method performance categories.

Signatures:

The printed name, signature and date of each analyst involved in the initial demonstration of method performance is to be provided at the bottom of the checklist sheet.

~~C.2~~ E.1.3 EPA PBMS Checklist for Continuing Demonstration of Capability:

The process by which a laboratory documents that its previously established performance of an analytical

procedure continues to meet performance specifications as delineated in this checklist.

#1. Method Blank Result.

A clean matrix (i.e., does not contain the analytes of interest) that is carried through the entire analytical procedure, including all sample handling, preparation, extraction, digestion, cleanup and instrumental procedures. The volume or weight of the blank should be the same as that used for sample analyses. The method blank is used to evaluate the levels of analytes that may be introduced into the samples as a result of background contamination in the laboratory. Enter the analyte/s and concentration measured in the blank.

#2. Concentrations of calibration standards used to verify working range, where applicable (include units).

The range of the concentration(s) of materials used to confirm the established relationship between the response of the measurement system and analyte concentration. This range should bracket any action, decision or regulatory limit. In addition, this range must include the concentration range for which sample results are measured and reported (when samples are measured after sample dilution/concentration). Enter the concentrations of the calibration standards.

#3. Calibration Verification.

A means of confirming that the previously determined calibration relationship still holds. This process typically involves the analyses of two standards with concentrations which bracket the concentration(s) measured in the sample/s. Enter the procedure to be used to verify the calibration and the results obtained for each analyte.

#4. Laboratory Control Sample.

An analytical standard carried through all aspects of the analytical method, e.g., digestions, distillations and determinative steps/instrumentation. It is generally used to assess the performance of all of the measurement system independent of the challenges of the sample matrix.

#5. External QC sample (where applicable).

Enter the results of analyses for reference material (e.g., quality control samples/ampoules) from a source different from that used to prepare calibration standards (where applicable). Enter the concentration, as well as, the source of this material. This performance category is of particular importance if Performance Evaluation (PE) studies are not available for the analytes of interest.

#6. Performance Evaluation Studies performed for analytes of interest, where available (last study sponsor and title last study number:).

Several EPA programs conduct periodic performance evaluation (PE) studies. Organizations outside of the Agency also may conduct such studies. Where available and applicable, enter the sponsor, title, and date of the most recent study in which the performance-based method was applied to the matrix of interest. A program/project may specify that a performance-based method be fully successful, i.e., within the PE study QC acceptance criteria.

7. List of analytes for which results were “not acceptable” in PE study where available and applicable..

#8. Surrogates used, if applicable.

Enter the names of the surrogate compounds used. Surrogates are often used in analysis of organic analytes. Surrogates may be added to samples prior to preparation, as a test of the entire analytical procedure. These compounds are typically brominated, fluorinated or isotopically labeled, with structural similarities to the analytes of interest. Target analytes of the method may be used as surrogates, if they can be demonstrated not to be present in the samples to be analyzed.

#9. Concentration of surrogates, if applicable.

Enter the concentration of surrogates once spiked into the sample (i.e., final concentration), with units.

#10. Recoveries of Surrogates appropriate to the proposed use (if applicable).

Enter the summary of the surrogate recovery limits and attached a detailed listing (each surrogate compound), if more space is needed.

#11. Matrix (reagent water, drinking water, sand, loam, clay, waste solid, ambient air, etc.).

Refers to the specific sample type within the broader "Medium" that was spiked, e.g., for Medium: Waste an example matrix, spiked as part of the initial demonstration of method performance, might be solvent waste.

#12. Matrix Spike Compounds.

Enter the analytes spiked. In preparing a matrix spike, a known amount of analyte is added to an aliquot of a real-world sample matrix. This aliquot is analyzed to help evaluate the effects of the sample matrix on the analytical procedure. Matrix spike results are typically used to calculate recovery of analytes as a measure of bias for that matrix.

#13. Matrix Spike Concentrations (w/units corresponding to final sample concentration).

Enter the amount of the analyte/s or "spike" that was added to the sample matrix in terms of the final concentration in the sample.

#14. Recovery of Matrix Spike (w/units).

The ratio of the standard deviation of a series of at least three measurements to the mean of the measurements. This value is often expressed as a percentage of the mean.

Note: Some programs/projects have utilized matrix spike duplicates (a separate duplicate of the matrix spike) to help verify the matrix spike result and to provide precision data for analytes which are not found in real-world samples, since duplicates of non-detects provides little information concerning the precision of the method. See Item # 19.

#15. Qualitative identification criteria used.

Enter all relevant criteria used for identification, including such items as retention times, spectral wavelengths, and ion abundance ratios. If the instrumental techniques for the performance-based method are similar to a reference method, use the reference method as a guide when specifying identification criteria. If the list of criteria is lengthy, attach it on a separate sheet, and enter "see attached" for this item.

#16. Precision (analyte by analyte).

#17-18. Other category.

Enter other necessary program/project specific method performance categories.

Signatures:

The printed name, signature and date of each analyst involved in the initial demonstration of method performance is to be provided at the bottom of the checklist sheet.

EPA Performance-Based Measurement System Certification Statement

Date: **Page __of __**

Laboratory Name & Address

Facility Name:

Discharge Point ID, where applicable:

EPA Program and Applicable Regulation:

Medium:

(i.e., water, soil, air, waste solid, leachate, sludge, other)

Analyte, Class of Analytes or Measured Parameters (CAS # where available)

(i.e , barium, trace metals, benzene, volatile organics, etc.)

We, the undersigned, CERTIFY that:

1. The methods in use at this facility for the analyses of samples for the programs of the U.S. Environmental Protection Agency have met the Initial and any required Continuing Demonstration of Method Performance Criteria specified under the Performance-Based Measurement System.

2. A copy of the Performance-Based Method, written in EMMC format, and copies of the reference method and laboratory-specific SOPs are available for all personnel on-site.

3. The data and checklists associated with the initial and continuing demonstration of method performance are true, accurate, complete and self-explanatory (1).

4. All raw data (including a copy of this certification form) necessary to reconstruct and validate these performance related analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized inspectors.

_____	_____	_____
Facility Manager's Name and Title	Signature	Date
_____	_____	_____
Quality Assurance Officer's Name	Signature	Date

This certification form must be completed when the performance-based method is originally certified, each time a continuing demonstration of method performance is documented, and whenever a change of personnel involves the Facility Manager or the Quality Assurance Officer.

(1) True: Consistent with supporting data.

Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.

Complete: Includes the results of all supporting performance testing.

Self-Explanatory: Data properly labeled and stored so that the results are clear and require no additional explanation.

EPA PBMS
Checklist for Initial Demonstration of Method Performance

***Provide a checklist <u>for each matrix</u> included in the demonstration*.**

Date: **Page __of __**
Laboratory Name & Address:
Facility Name:
Discharge Point ID, where applicable:
EPA Program and Applicable Regulation:
Medium:
(i.e., water, soil, air, waste solid, leachate, sludge, other)
Analyte, Class of Analytes or Other Measured Parameters (CAS #, where available):
(i.e., barium, trace metals, benzene, volatile organics, etc.)

Initial Demonstration of Method Performance *(1)*				
Category	**Performance Criteria *(2)* Based on**		**Results Obtained**	**Perf. Spec. Achieved (✓)**
	Reference Method	**Measurement Quality Objective**		
1. Written method (addressing all elements in the EMMC format) attached				
2. Title, number and date/rev. of "reference method", if applicable ***(3)***				
3. Copy of the reference method, if applicable, maintained at facility				
4. Differences between PBM and reference method (if applicable) attached				
5. Concentrations of calibration standards				
6. %RSD or slope/correlation coefficient of calibration regression				
7. Performance range tested (with units)				
8. Sample(s) used in initial demonstration have recommended				

Initial Demonstration of Method Performance *(1)*				
Category	**Performance Criteria *(2)* Based on**		**Results Obtained**	**Perf. Spec. Achieved (✓)**
	Reference Method	**Measurement Quality Objective**		
9. Samples(s) used in initial demonstration met recommended holding times, where applicable				
10. Interferences				
11. Qualitative identification criteria used				
12. Performance Evaluation studies performed for analytes of interest, where available: Last study sponsor and title: Last study number:				
13. Analysis of external reference material Last study sponsor and title: Last study number: List of analytes with “not acceptable” results:				
14. Source of reference material				
15. Surrogates used, if applicable				
16. Concentrations of surrogates, if applicable				
17. Recoveries of Surrogates appropriate to the proposed use, if applicable				
18. Sample preparation				
19. Clean-up procedures				
20. Method Blank Result				
21. Matrix (reagent water, drinking water, sand, waste solid, ambient air, etc.)				
22. Spiking system, appropriate to method and application				
23. Spike concentrations (w/ units corresponding to final sample concentration)				

Initial Demonstration of Method Performance *(1)*				
Category	**Performance Criteria *(2)* Based on**		**Results Obtained**	**Perf. Spec. Achieved (✓)**
	Reference Method	**Measurement Quality Objective**		
24. Source of spiking material				
25. Number of replicate spikes				
26. Precision (analyte by analyte)				
27. Bias (analyte by analyte)				
28. Detection Limit (w/ units; analyte by analyte)				
29. Confirmation of Detection Limit, if applicable				
30. Quantitation Limit (w/ units: analyte by analyte)				
31. Qualitative Confirmation				
32. Frequency of performance of the Initial Demonstration				
33. Other criterion (specify)				
34. Other criterion (specify)				

[1] Provide a detailed narrative description of the initial demonstration.

[2] For multi-analyte methods, enter "see attachment" and attach a list or table containing the analyte-specific performance criteria from the reference method or those needed to satisfy measurement quality objectives.

[3] If a reference method is the source of the performance criteria, the reference method should be appropriate to the required application, and the listed criteria should be fully consistent with that reference method.

Name and signature of each analyst involved in the initial demonstration of method performance (includes all steps in the proposed method/modification):

_____	_____	_____
Name	**Signature**	**Date**
_____	_____	_____
Name	**Signature**	**Date**
_____	_____	_____
Name	**Signature**	**Date**

The certification above must accompany this form each time it is submitted.

EPA PBMS
Checklist for Continuing Demonstration of Method Performance

Date: **Page __of __**
Facility Name:
Laboratory Name & Address:
Discharge Point ID, where applicable:
EPA Program and Applicable Regulation:
Medium:
(i.e.,water, soil, air, waste solid, leachate, sludge, other)
Analyte, Class of Analytes or Measured Parameters (CAS # where available)
(i.e., barium, trace metals, benzene, volatile organics, etc.)

Continuing Demonstration of Method Performance				
Category	Required Frequency	Specific Performance Criteria	Results Obtained	Perf. Spec. Achieved (✓)
1. Method blank result (taken through all steps in the procedure)				
2. Concentrations of calibration standards used to verify working range (with units), where applicable				
3. Calibration verification				
4. Laboratory Control Sample				
5. External QC sample (where available)				
6. Performance evaluation (PE) studies, if applicable Last study sponsor and title: Last study number:				
7. List analytes for which results were "not acceptable" in PE study	----	----	----	----
8. Surrogates used, if applicable				
9. Concentration of Surrogates, if applicable				
10. Recovery of Surrogates (acceptance range for multianalyte methods), if applicable				
11. Matrix				
12. Matrix spike compounds				
13. Concentration of Matrix spike compounds				
14. Recoveries of Matrix spike compounds				
15. Qualitative identification criteria used				
16. Precision (analyte by analyte)				
17. Other category (specify)				
18. Other category (specify)				

EPA PBMS
Checklist for Continuing Demonstration of Method Performance

Date: **Page __of __**

Facility Name:

Discharge Point ID, where applicable:

EPA Program and Applicable Regulation:

Medium:
(i.e. water, soil, air, waste solid, leachate, sludge, other)

Analyte, Class of Analytes or Measureand (CAS # where available)
(i.e. barium, trace metals, benzene, volatile organics, etc.)

Name and signature of each analyst involved in continuing demonstration of method performance (includes all steps in the proposed method/modification):

_____	_____	_____
Name	**Signature**	**Date**
_____	_____	_____
Name	**Signature**	**Date**
_____	_____	_____
Name	**Signature**	**Date**

The certification above must accompany this form each time it is submitted.